International Standard 1386

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Solvent acetates for industrial use — Methods of test

Acétates solvants à usage industriel — Méthodes d'essai

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 1386 was developed by Technical Committee ISO/TC 47, *Chemistry*. It results from the combination into one single document of draft International Standards ISO/DIS 1386/1, ISO/DIS 1386/2 and ISO/DIS 1386/3, which were circulated to the member bodies in September 1982 and have been approved by the member bodies of the following countries:

Australia
Austria
Belgium
Bulgaria
China (DIS 1386/2)
Czechoslovakia
Egypt, Arab Rep. of
France
Germany, F.R.
Hungary
India
Italy
Korea, Rep. of
Netherlands
  (DIS 1386/2 and
  DIS 1386/3)
New Zealand
  (DIS 1386/3)
Nigeria
Poland
Portugal
Romania
South Africa, Rep. of
Switzerland

The member body of the following country expressed disapproval of ISO/DIS 1386/1 on technical grounds:

Netherlands

No member body expressed disapproval of parts 2 and 3 of the draft.

This International Standard cancels and replaces ISO Recommendation R 1386-1970, of which it constitutes a technical revision.

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Solvent acetates for industrial use — Methods of test

1 Scope and field of application

This International Standard gives general instructions and specifies methods of test for the analysis of solvent acetates most frequently used in industry.

The methods of test relating to solvent acetates for industrial use are the following:

- Determination of density at 20 °C
- Determination of dry residue after evaporation on a water bath
- Measurement of colour
- Determination of distillation characteristics
- Determination of water content
- Determination of acidity — Titrimetric method
- Determination of ester content — Titrimetric method after saponification

2 References

ISO 758, *Liquid chemical products for industrial use — Determination of density at 20 °C.*

ISO 759, *Volatile organic liquids for industrial use — Determination of dry residue after evaporation on a water bath — General method.*

ISO 760, *Determination of water — Karl Fischer method (General method).*

ISO 918, *Volatile organic liquids for industrial use — Determination of distillation characteristics.*

ISO 2211, *Liquid chemical products — Measurement of colour in Hazen units (platinum-cobalt scale).*

General instructions

3 Sampling[1]

Store the laboratory sample in a clean, dry and air-tight bottle fitted either with a ground glass stopper or with a screw cap containing an air-tight inert plastics seal. The bottle should be of such capacity that it is almost entirely filled by the sample. If it is necessary to seal the container, take care to avoid any contamination of the contents.

NOTE — A sample of not less than 500 ml is necessary for performing all the tests specified for these products.

4 Test report

The test report, for each determination, shall contain the following particulars:

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

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[1] The sampling of liquid chemical products for industrial use will form the subject of a future International Standard.

Methods of test

5 Determination of density at 20 °C

Use the method specified in ISO 758.

6 Determination of dry residue after evaporation on a water bath

Use the method specified in ISO 759.

7 Measurement of colour

Use the method specified in ISO 2211.

8 Determination of distillation characteristics

Use the method specified in ISO 918, subject to the following details appropriate to solvent acetates.

8.1 Thermometer (see ISO 918, sub-clause 5.1.2) covering a range suitable for the product under test (see table 1).

8.2 Temperature correction

Calculate this correction using the appropriate formula given in the table and add it algebraically to the result (see ISO 918, sub-clause 9.2.2).

9 Determination of water content

Use the method specified in ISO 760.

Table 1

Product	Thermometer scale covering range[1]	Correction to be applied to temperatures[2]	Interval before the first drop of distillate falls from the end of the condenser
	°C	°C	min
Methyl acetate	24 to 78	$0{,}030\ (1\ 013 - p)$	7 to 12
Ethyl acetate	48 to 102	$0{,}031\ (1\ 013 - p)$	7 to 12
Isopropyl acetate	72 to 126	$0{,}032\ (1\ 013 - p)$	7 to 12
Isobutyl acetate	98 to 152	$0{,}034\ (1\ 013 - p)$	10 to 15
Butyl acetate	98 to 152	$0{,}037\ (1\ 013 - p)$	10 to 15
Isopentyl and pentyl acetates	123 to 177	$0{,}038\ (1\ 013 - p)$	10 to 15

1) Or any other suitable scale. The temperature intervals, given as a guide, are taken from the standard ASTM E 1.

2) p is the atmospheric pressure, in millibars. If the pressure p is measured in pascals (or in kilopascals), multiply the value by 0,01 (or by 10) to convert it to millibars.

The factors by which the corrected values of atmospheric pressure are multiplied represent the coefficients of variation of the distillation point of products given as a function of atmospheric pressure, in millibars.

10 Determination of acidity — Titrimetric method

10.1 Principle

Dilution of test portion in 95 % (V/V) ethanol and titration of the acidity using a standard volumetric sodium hydroxide solution, in the presence of an indicator. Two cases are specified:

- methyl and ethyl acetates: titration of the acidity at 0 °C, with phenol red as indicator;
- isopropyl, isobutyl, butyl, isopentyl and pentyl acetates: titration of the acidity at room temperature, with phenolphthalein as indicator.

10.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

10.2.1 Ethanol, 95 % (V/V).

10.2.2 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0{,}1$ mol/l.

10.2.3 Phenol red, 0,2 g/l ethanolic solution.

Warm 0,05 g of phenol red with 2,85 ml of a 2,0 g/l sodium hydroxide solution and 5 ml of the ethanol (10.2.1). When dissolution is complete, add 50 ml of the same ethanol and dilute to 250 ml with water.

10.2.4 Phenolphthalein, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 100 ml of the ethanol (10.2.1) and make faintly pink by the addition of the standard volumetric sodium hydroxide solution (10.2.2).

10.3 Apparatus

Ordinary laboratory apparatus, and

10.3.1 Conical flask, of borosilicate glass, capacity 250 ml, fitted with a stopper carrying a soda-lime tube.

10.3.2 Microburette, capacity 10 ml, graduated in 0,02 ml divisions or 0,01 ml for acidities, expressed as a percentage by mass of acetic acid, of less than 0,012 %.

10.4 Procedure

10.4.1 Methyl and ethyl acetates

10.4.1.1 Test portion

Weigh, to the nearest 0,05 g, a mass of between 50 and 100 g of the laboratory sample, according to the expected acidity and, at the same time, to maximize the sensitivity of the method.

10.4.1.2 Determination

Place 50 ml of the ethanol (10.2.1) in the conical flask (10.3.1), add a few drops of the phenol red solution (10.2.3) and make just red by addition of the standard volumetric sodium hydroxide solution (10.2.2). Stopper the flasks and cool to 0 °C. Withdraw the stopper, add the test portion (10.4.1.1), mix and titrate the mixture at 0 °C with the standard volumetric sodium hydroxide solution (10.2.2) until the red colour persists for at least 5 s.

10.4.2 Isopropyl, isobutyl, butyl, isopentyl and pentyl acetates

10.4.2.1 Test portion

Weigh, to the nearest 0,05 g, 50 to 100 g of the laboratory sample according to the expected acidity, expressed as acetic acid, to increase the sensitivity of the method.

10.4.2.2 Determination

Place 50 ml of the ethanol (10.2.1) in the conical flask (10.3.1), add 0,5 ml of the phenolphthalein solution (10.2.4) and make faintly pink by addition of the standard volumetric sodium hydroxide solution (10.2.2). Add the test portion (10.4.2.1), mix and titrate the mixture with the standard volumetric sodium hydroxide solution (10.2.2) until the pink colour persists for at least 5 s.

10.5 Expression of results

The acidity, expressed as a percentage by mass of acetic acid (CH_3COOH), is given by the formula

$$\frac{0{,}006 \times V \times 100}{m}$$

$$= \frac{0{,}6\,V}{m}$$

where

V is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (10.2.2), used for the determination;

m is the mass, in grams, of the test portion (10.4.1.1 or 10.4.2.1);

0,006 is the mass, in grams, of acetic acid corresponding to 1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 0{,}100$ mol/l.

NOTE — If the concentration of the sodium hydroxide solution (10.2.2) is not exactly as specified in the list of reagents, an appropriate correction should be applied.

11 Determination of ester content — Titrimetric method after saponification

11.1 Principle

Saponification of the esters present in a test portion with excess ethanolic potassium hydroxide solution, and determination of the excess by titration with standard volumetric hydrochloric acid solution, using phenolphthalein as indicator.

11.2 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

11.2.1 Potassium hydroxide, 56 g/l solution in 95 % (V/V) ethanol.

11.2.2 Hydrochloric acid, standard volumetric solution, $c(\text{HCl}) = 1$ mol/l.

11.2.3 Phenolphtalein, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 100 ml of 95 % (V/V) ethanol and make faintly pink by addition of dilute sodium hydroxide solution.

11.3 Apparatus

Ordinary laboratory apparatus, and

11.3.1 Two conical flasks, of borosilicate glass, capacity 250 ml, fitted with ground glass stoppers.

11.3.2 Reflux condensers, water-cooled, with ground glass joints to fit the conical flasks (11.3.1).

11.3.3 Weighing pipette, capacity 10 ml.

11.4 Procedure

11.4.1 Test portion and blank test

Introduce successively, from the same one-mark pipette 50,0 ml of the potassium hydroxide solution (11.2.1) into each of the two conical flasks (11.3.1).

Using the weighing pipette (11.3.3), transfer immediately, weighed to the nearest 0,000 1 g, the test portion specified in table 2 into one of the flasks.

Table 2

Product	Alkyl radical (R)	Mass of test portion (m)	Relative molecular mass
		g	(M_r)
Methyl acetate	CH_3	1,6 to 2,1	74,1
Ethyl acetate	C_2H_5	2,0 to 2,4	88,1
Isopropyl acetate	C_3H_7	2,4 to 2,8	102,1
Butyl acetates	C_4H_9	2,7 to 3,2	116,2
Isopentyl and pentyl acetates	C_5H_{11}	3,0 to 3,5	130,2

11.4.2 Determination

11.4.2.1 Methyl and ethyl acetates

Close the two flasks, one of which contains the test portion, with their ground-glass stoppers and leave to stand at ambient temperature (not less than 15 °C) for 4 h. Add 0,5 ml of the phenolphthalein solution (11.2.3) to each flask and immediately titrate first the test solution and then the blank test solution with the standard volumetric hydrochloric acid solution (11.2.2) until the pink colour is discharged.

11.4.2.2 Isopropyl, butyl, isopentyl and pentyl acetates

Heat the two flasks, one of which contains the test portion, fitted with the reflux condensers (11.3.2), for 1 h in a boiling-water bath. Withdraw the flasks, still carrying their condensers, and immerse them in cold running water. When cold, wash down the inside of each condenser with two 20 ml portions of water, collecting the washings in the flasks. Disconnect the flasks and wash each joint with a further 20 ml of water again collecting the washings in the appropriate flasks.

Add 0,5 ml of the phenolphthalein solution (11.2.3) to each flask and immediately titrate first the test solution and then the blank test solution with the standard volumetric hydrochloric acid solution (11.2.2) until the pink colour is discharged.

11.5 Expression of results

The ester content, expressed as a percentage by mass of the acetate CH_3COOR (where **R** is the appropriate alkyl radical specified in table 2), is given by the formula

$$\frac{M_r(V_0 - V_1)}{1\,000\,m} \times 100 - \frac{M_r \times A}{60}$$

$$= \frac{M_r(V_0 - V_1)}{10\,m} - \frac{M_r \times A}{60}$$

where

M_r is the relative molecular mass of the acetate (see table 2);

V_0 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (11.2.2), used for the blank test;

V_1 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (11.2.2), used for the determination;

m is the mass, in grams, of the test portion;

A is the acidity, expressed as a percentage by mass of acetic acid, determined using the test method specified in clause 10;

60 is the relative molecular mass of acetic acid.

NOTE — If the concentration of the hydrochloric acid solution (11.2.2) is not exactly as specified in the list of reagents, an appropriate correction should be applied.